

1007-020

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE  
PATENT OPERATION

In re application of:

Hidenobu Mikami et al.

Serial No.: 10/624,002

Group Art Unit: 1714

Filed: July 21, 2003

Examiner: Poulos, Sandra K.

For: A GREASE COMPOSITION HAVING A BASE OIL, A THICKENER, AND AN ADDITIVE  
INCLUDING A METAL SALT OF DIBASIC ACIDCommissioner for Patents  
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## DECLARATION UNDER 37 CFR 1.132

Sir:

I, Hidenobu Mikami, a named inventor in the above identified application, have personally supervised the following tests:

## 1. Comparative Example 5

Alkyldiphenyl ether oil as a base oil was prepared in the amount shown in the attached Table 2a. The base oil is separated into three separate liquid portions. Separately, p-toluidine, cyclohexyl amine and 4,4'-diphenyl methane diisocyanate (MDI) were separately dissolved in one of the three separate liquid portions prepared above in an amount recorded in Table 2a. While the MDI, dissolved in the base oil, was stirred, the other solutions of the p-toluidine and cyclohexyl amine were added to the solution of the MDI. The mixed solution was stirred at 100 to 120°C for 30 minutes. Ba sulfonate, sorbitantriolate and an antioxidant were added thereto in the amount shown in Table 2a and stirred at 100 to 120°C for 10 minutes. After cooling, the mixed solution was homogenized using a three-roll mixer to provide a grease composition. This grease composition was evaluated using a high temperature and high speed test, a quick acceleration and deceleration test and a rust preventative test. The results are shown in Table 2a.

A rust preventative test was conducted in accordance with, but under more severe test conditions than the method set forth in ASTM D1743. A conical roller bearing "30204" was degreased with an organic solvent and dried. The bearing was sealed with 1.9 to 2.1g of the grease composition being tested and a 98N axial load was applied to the bearing. Running-in was performed for 1 minute at 1800 rpm. Thereafter, the bearing was dipped with a 1% by weight of saline solution and introduced into a sealed high humidity vessel where it reached a saturated vapor pressure at 40°C and allowed to stand for 48 hours at 40°C. Rust generation was observed and determined by counting the number of positions where rust was generated. An outer ring of the bearing was divided into 32 portions for purposes of counting the numbers of the positions where rust was generated.

Results are reported as the average of four tests, and are shown in Table 2a.

Table 2a

Component (parts by weight)	Comparative Example			
	5	6	7	8
<b>Base oil</b>				
Synthesized hydrocarbon oil <sup>(1)</sup>	-	-	-	16
Alkyldiphenyl ether oil <sup>(2)</sup>	80	78	83	64
<b>Thickeners</b>				
Amine, p-toluidine	4.6	4.6	-	9.3
Amine, Cyclohexyl amine	4.6	-	-	-
Amine, Octyl amine	-	5.1	8.6	-
Diisocyanate, MDI <sup>(3)</sup>	11.0	10.3	8.4	10.7
<b>Additives</b>				
Na sulfonate	1	1	1	1
Sorbitanester <sup>(4)</sup>	1	1	1	-
Metal salt of dibasic acid (Na sebacate)	-	-	-	-
Sodium nitrite	-	-	-	1
<b>Antioxidant</b>				
Alkylated diphenylamine	2	2	2	2
Dilaurylthiodipropionate	-	-	-	-
Tetrakis-(methylene-3-(3',5'-di- <i>t</i> -butyl-4-hydroxyphenyl)propionate)methane	-	-	-	-
<b>Properties</b>				
Viscosity of base oil (40°C, mm <sup>2</sup> /s)	97	97	97	72
Worked penetration (VTS K2220)	285	280	260	290
High temperature and high speed test, h	1200	1300	600	400
Quick acceleration and deceleration test, h	200	250	120	>300
Rust preventative properties, number	6	4	1	1

<sup>(1)</sup> "Shinfluid 601" from Nippon Steel Chemical Co., Ltd.

<sup>(2)</sup> "L100" from Matsumura Sekiyu KK

<sup>(3)</sup> diphenylmethanediisocyanate

<sup>(4)</sup> sorbitantriolate

## 2. Comparative Examples 6 to 8

In each example, thickeners and base oil were selected in a ratio shown in Table 2a in order to prepare a base grease in a manner similar to Comparative Example 5. An additive was added to the base grease to form the grease composition. In Comparative Example 6, p-toluidine, octyl amine and MDI were added as thickeners and in Comparative Example 7, octyl amine and MDI were added as thickeners. In Comparative Example 8, sodium nitrite was used as an additive. The resultant grease compositions were tested for the above-mentioned tests in a manner similar to Comparative Example 5. The results are also shown in Table 2a.

### High temperature and High Speed Test

A ball-and-roller bearing (6204) was scaled with 0.7g of grease to be tested and rotated at 10,000 rpm, at an outer diameter temperature of an outer ring of the bearing of 150°C under a radial load of 67N and an axial load of 67N. A time to seizure was measured. The results are shown in Table 2a.

### Quick Acceleration and Deceleration Test

A quick acceleration and deceleration test was conducted on a ball-and-roller bearing that supported a rotating shaft with an inner ring; the rotating shaft supporting a pulley around which a rotating belt of an alternator was wound; the alternator being one example of auxiliary electric equipment. Operating conditions for the quick acceleration and deceleration test were as follows: a load of 3234 N to the pulley; and a rotating speed of 0 to 18,000 rpm. An endurance time (life time) was measured as the time to produce abnormal peeling within the bearing, to detect higher vibration than a set value by an oscillation detector and finally a time to stop a generator. The results are shown in Table 2a.

### Rust Preventative Test

As shown in Table 2a, the grease compositions according to the above Comparative Examples provide inferior results as compared to one or more of the tests of the greases of Original Examples 1-8 of the above identified application. When Comparative Example 8 of Table 2a is compared with Original Example 7, which is made from the same base oil and has same amount of the same thickener and also has the same viscosity, the profound effect of the metal salt of the dibasic acid on the grease composition of the present invention is readily apparent. While the viscosity of both greases are the same, the results in the high temperature/high speed test of the composition according to the invention are such that the time to failure is about seven times longer for the grease composition which is a result that could not have been predicted based on the properties of prior art grease compositions.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application and of any patent issued thereon.

Dated: \_\_\_\_\_

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Hidenobu Mikami